



AFRL-RX-WP-TP-2009-4158

THE EFFECT OF HOLES ON THE RESIDUAL STRENGTH OF SiC/SiC CERAMIC COMPOSITES (PREPRINT)

**G. Ojard, Y. Gawayed, U. Santhosh, J. Ahmad, R. Miller, and R. John
Pratt & Whitney**

NOVEMBER 2007

Approved for public release; distribution unlimited.

See additional restrictions described on inside pages

STINFO COPY

**AIR FORCE RESEARCH LABORATORY
MATERIALS AND MANUFACTURING DIRECTORATE
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750
AIR FORCE MATERIEL COMMAND
UNITED STATES AIR FORCE**

REPORT DOCUMENTATION PAGE				Form Approved OMB No. 0704-0188	
<p>The public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.</p>					
1. REPORT DATE (DD-MM-YY) November 2007		2. REPORT TYPE Journal Article Preprint		3. DATES COVERED (From - To) 01 November 2007 – 01 November 2007	
4. TITLE AND SUBTITLE THE EFFECT OF HOLES ON THE RESIDUAL STRENGTH OF SiC/SiC CERAMIC COMPOSITES (PREPRINT)				5a. CONTRACT NUMBER F33615-03-D-2354-0004	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER 62102F	
6. AUTHOR(S) G. Ojard and R. Miller (Pratt & Whitney), Y. Gawayed (Auburn University), U. Santhosh and J. Ahmaad (Research Applications, Inc.) R. John (AFRL/RXLMN)				5d. PROJECT NUMBER 4347	
				5e. TASK NUMBER 53	
				5f. WORK UNIT NUMBER 43475314	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Pratt & Whitney 400 Main Street East Hartford, CT 06108				8. PERFORMING ORGANIZATION REPORT NUMBER Auburn University, Auburn, AL Research Applications, Inc., San Diego, CA	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Air Force Research Laboratory Materials and Manufacturing Directorate Wright-Patterson Air Force Base, OH 45433-7750 Air Force Materiel Command United States Air Force				10. SPONSORING/MONITORING AGENCY ACRONYM(S) AFRL/RXLMN	
				11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S) AFRL-RX-WP-TP-2009-4158	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited.					
13. SUPPLEMENTARY NOTES To be submitted to International Journal of Plasticity PAO Case Number and clearance date: WPAFB-07-0725, 17 December 2007. The U.S. Government is joint author of this work and has the right to use, modify, reproduce, release, perform, display, or disclose the work.					
14. ABSTRACT Continued interest in ceramic matrix composites for the use in high temperature applications necessitates a comprehensive understanding of the effect of time-dependent loading in various environments on the material mechanical response. This understanding has to include the effect of structural features, such as holes, on the material performance. In this work, residual strength tests were conducted for samples with 2.286, and 4.572 mm diameter holes, located at the center of the specimen, after time-dependent experiments conducted in air. Samples were subjected to creep and dwell fatigue tests at 1204 degrees Celsius under net-section stresses ranging from 55.16 to 165.48 MPa for durations ranging from 10 to 2400 hours prior to residual strength experiments at room temperature. Data acquired from residual strength testing for samples with holes are analyzed and compared to similar data from standard samples.					
15. SUBJECT TERMS ceramic matrix composites, high temperature, time-dependent loading, strength test, dwell fatigue, creep fatigue					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT: SAR	18. NUMBER OF PAGES 14	19a. NAME OF RESPONSIBLE PERSON (Monitor) Reji John 19b. TELEPHONE NUMBER (Include Area Code) N/A
a. REPORT Unclassified	b. ABSTRACT Unclassified	c. THIS PAGE Unclassified			

THE EFFECT OF HOLES ON THE RESIDUAL STRENGTH OF SiC/SiC CERAMIC COMPOSITES

Ojard, G.², Gowayed, Y.³, Santhosh, U.⁴, Ahmad J.⁴, Miller, R.², and John, R.¹

¹ Air Force Research Laboratory, AFRL/MLLMN, Wright-Patterson AFB, OH

² Pratt & Whitney, East Hartford, CT

³ Auburn University, Auburn, AL

⁴ Research Applications, Inc., San Diego, CA

ABSTRACT

Continued interest in ceramic matrix composites for the use in high temperature applications necessitates a comprehensive understanding of the effect of time-dependent loading in various environments on the material's mechanical response. This understanding has to include the effect of structural features, such as holes, on the material performance. In this work, residual strength tests were conducted for samples with 2.286, and 4.572 mm average diameter holes, located at the center of the specimen, after time-dependent experiments conducted in air. Samples were subjected to creep and dwell fatigue tests at 1204 °C under net-section stresses ranging from 55.16 to 165.48 MPa for durations ranging from 10 to 2400 hours prior to residual strength experiments at room temperature. Data acquired from residual strength testing for samples with holes are analyzed and compared to similar data from standard samples.

INTRODUCTION

Testing is conducted to understand the long-term behavior of Ceramic Matrix Composites (CMCs) under conditions of sustained load at high temperatures. Examples of long-term environments would include ground base turbines for power generation where CMCs are being considered for combustor liners, turbine vanes and shroud applications. These applications can see design times of up to 30,000 hours. Such long term applications are working to leverage the high temperature material capability while taking advantage of reduced cooling and durability improvements that CMCs can provide over typical metals without cooling air that is needed for the nickel base superalloys as a possible replacement. This paper reports data and analysis of specimens with holes subjected to time-dependent tests followed by a residual strength experiments conducted at room temperature and compared to data on standard specimens published in previous years.

EXPERIMENTAL PROGRAM

Materials and Manufacturing

The material chosen for the study was the Melt Infiltrated SiC/SiC CMC system, which was initially developed under the Enabling Propulsion Materials Program (EPM) and is still under further refinement at NASA-Glenn Research Center (GRC). This material system has been systematically studied at various development periods and the most promising was the 01/01 Melt Infiltrated iBN SiC/SiC (01/01 is indicative of the month and year that development was frozen). There is a wide set of data from NASA for this system as well as a broad historic database from the material development. This allowed a testing system to be put into place to look for key development properties which would be needed from a modeling effort and would hence leverage existing data generated by NASA-GRC.

The Sylramic[®] fiber was fabricated by DuPont as a 10 μm diameter stoichiometric SiC fiber and bundled into tows of 800 fibers each. The sizing applied was polyvinyl alcohol (PVA). For this study, the four lots of fibers, which were used, were wound on 19 different spools. The tow spools were then woven into a 5HS balanced weave at 20 EPI. An in-situ Boron Nitride (iBN) treatment was performed on the weave (at NASA-GRC), which created a fine layer of BN on every fiber. The fabric was then laid in graphite tooling to correspond to the final part design (flat plates for this experimental program). All the panels were manufactured from a symmetric cross ply laminate using a total of 8 plies. The graphite tooling has holes to allow the CVI deposition to occur. At this stage, another BN coat layer was applied. This BN coating was doped with Si to provide better environmental protection of the interface. This was followed by SiC vapor deposition around the tows. Typically, densification is done to about 30% open porosity. SiC particulates are then slurry cast into the material followed by melt infiltration of a Si alloy to arrive at a nearly full density material. The material at this time has less than 2% open porosity.

After fabrication, all the panels were interrogated by pulse echo ultrasound (10 MHz) and film X-ray. There was no indication of any delamination and no gross porosity regions were noted in the panels. In addition, each panel had two tensile bars extracted for witness testing at room temperature. All samples tested failed above a 0.3% strain to failure requirement. Hence, all panels were accepted into the testing effort. Samples were cut out of the accepted plates and holes were drilled in diameters of 2.286 and 4.572 mm at the center of the specimens forming 20% of the width of the specimen.

TESTING PROCEDURE

Creep testing at 1204 °C (exposure)

Creep testing is a test to determine the strain-time dependence of a material under a constant load. This test is also used to determine the long-term behavior of the material under combination of load and temperature. For ceramic matrix composites, when this testing is done in air, there is an added complexity of environmental exposure. A total of 12 creep tests were done on straight-sided tensile bars with two different central hole sizes (2.286 and 4.572 mm) at 1204 °C.

Dwell Fatigue at 1204 °C (exposure)

Dwell fatigue testing, sometimes referred to as “low cycle fatigue tests”, is the superposition of a stress hold at the peak stress of a fatigue cycle. Dwell fatigue tests were conducted for a dwell time of 2 hours with a 1 minute load and unload at the beginning and end of each cycle. Eighteen straight-sided tensile bars with two different central hole sizes (2.286 and 4.572 mm) were tested under 55.16, 110.32, and 165.48 MPa net-section stress levels at an R ratio of 0.05 at 1204 °C. Electronic data acquired were large in size and include load-reload tests at different time intervals. A Matlab code was developed to extract this data and allow characterization of the change in strain with time.

Residual strength tests at room temperature (post exposure)

Samples subjected to time dependent testing in creep or dwell fatigue experiments (exposure) were subjected to cyclic tensile loading at room temperature until failure (post exposure). Elastic and creep strains from exposure experiments as well as strain to failure during post-exposure

experiments were recorded along with the slope of the stress strain curves for apparent modulus calculations.

RESULTS AND DISCUSSION

Strain, stress to failure and modulus values acquired for the 30 specimens with holes are listed in Table 1 along with time-dependent strain and exposure time that each specimen witnessed during exposure testing. The time under exposure varied from 10 hours to 2400 hours. Sample of stress strain diagrams is shown in Figure 1 and 2.

Table 1: Experimental data for samples tested in residual strength after dwell fatigue and creep experiments at different net section stress levels:

Dwell Fatigue or Creep	Net Section Stress (MPa)	Time (hours)	Dwell Fatigue		Creep Strain During Exposure (mm/mm)	Residual			Apparent Re-Load Modulus (13-56 MPa) (GPa)
			Elastic	Max Strain		Apparent Modulus (13-56 MPa) (GPa)	UTS Net Section (MPa)	Strain to Failure (mm/mm)	
			Strain (loading) (mm/mm)	During Exposure (mm/mm)					
2.286 mm hole									
Dwell	55.16	10	0.000189	0.000305		313.7	349.58	0.0018	276.9
Dwell	55.16	100	0.000206	0.000427		298.3	420.60	0.0027	275.5
Dwell	55.16	10	0.000205	0.000298		309.3	379.23	0.0023	276.6
Dwell	55.16	100	0.000216	0.00038		309.8	392.33	0.0025	270.8
Dwell	165.48	500	0.000664	0.001648		292.5	336.92	0.0015	287.9
Dwell	165.48	524	0.000588	0.001259		302.2	325.85	0.0013	293.9
Dwell	165.48	436	0.000583	0.001665		288.2	302.40	0.0012	285.8
Dwell	110.32	100	0.000391	0.00134		301.4	341.30	0.0017	289.2
Dwell	110.32	10	0.000328	0.000519		301.6	372.33	0.0020	289.5
Dwell	110.32	100	0.000341	0.000591		314.4	404.74	0.0023	296.5
Creep	110.32	1004			0.0026	284.0	357.85	0.0023	262.9
Creep	55.16	2150			0.00293	261.9	264.21	0.0018	na
Creep	55.16	1129			0.00193	270.7	312.05	0.0022	228.6
Creep	110.32	1061			0.00163	270.5	368.19	na	245.1
Creep	55.16	2400			0.00387	259.7	309.90	0.0023	221.3
Creep	55.16	998			0.00078	279.0	325.95	0.0023	231.9
4.572 mm hole									
Dwell	110.32	100	0.00041	0.000824		289.2	335.10	0.0015	288.8
Dwell	110.32	10	0.000412	0.000546		303.9	330.96	0.0016	297.6
Dwell	110.32	100	0.000416	0.000637		303.5	343.37	0.0018	284.9
Dwell	55.16	10	0.000168	0.000125		304.7	346.82	0.0019	286.7
Dwell	55.16	100	0.000181	0.000351		293.0	341.30	0.0019	269.7
Dwell	110.32	10	0.000355	0.0007		300.4	330.96	0.0016	293.4
Dwell	55.16	100	0.000208	0.000364		316.4	363.19	0.0021	289.5
Dwell	55.16	10	0.000244	0.000293		316.5	352.33	0.0019	296.4
Creep	55.16	858			0.00373	286.3	314.41	0.0018	263.3
Creep	110.32	1041			0.00332	306.7	312.33	0.0015	287.6
Creep	55.16	1052			0.00113	297.0	282.14	0.0015	274.6
Creep	110.32	1046			0.00402	323.2	312.65	0.0014	306.1
Creep	110.32	1863			0.00515	279.5	210.61	0.0011	na
Creep	55.16	2065			0.00206	289.1	325.77	0.00183	261.5

na = not available

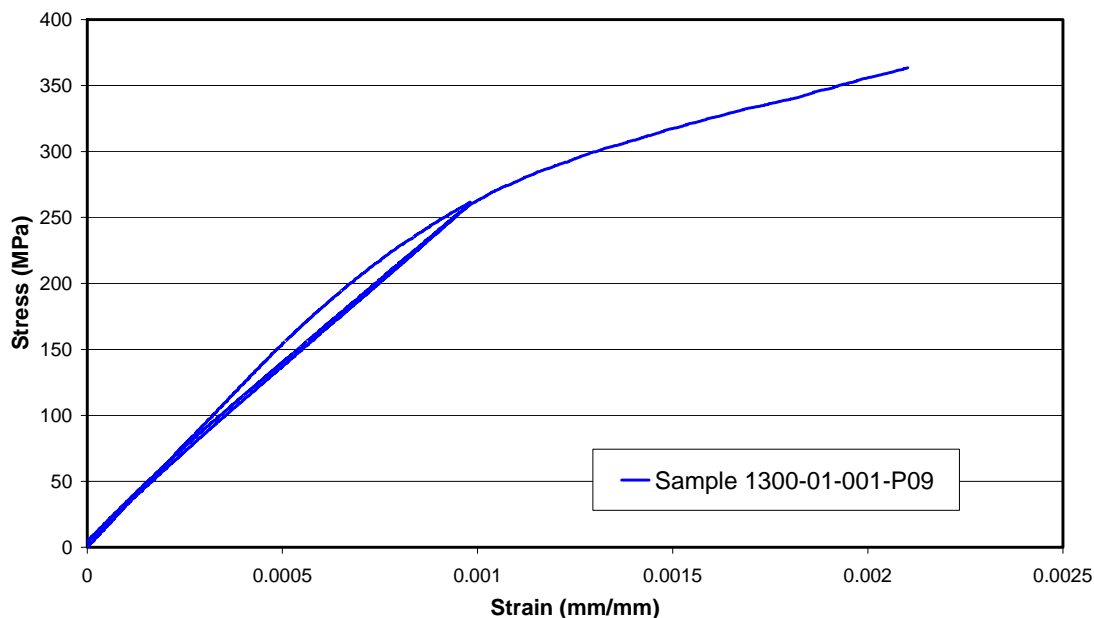


Figure 1. Residual strength for sample with 4.572 mm hole tested in dwell fatigue for 100 hours at 55.16 MPa

The effect of exposure time on residual strength is shown in Figure 3 and on the strain to failure is illustrated in Figure 4 along with data from standard samples as reference. Figure 5 shows the change of the value of strain to failure with the exposure strain for samples with and without holes.

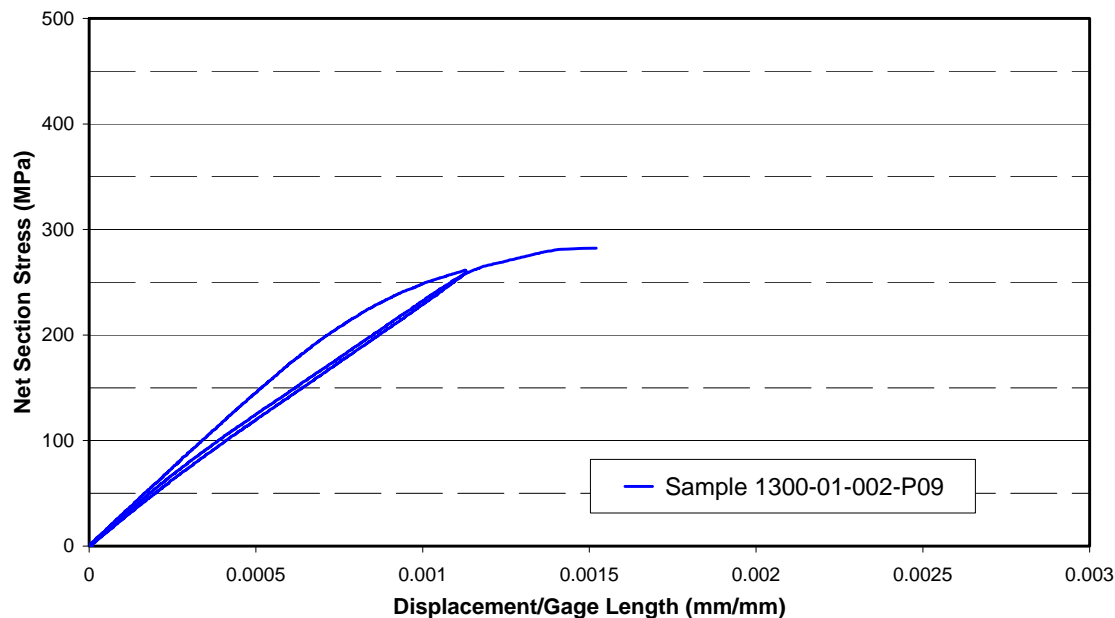


Figure 2. Residual strength for sample with 4.572 mm hole tested in dwell fatigue for 1052 hours at 55.16 MPa

It is important to note that the existence of the hole in the area of the gage length added an un-remediated artifact to the strain calculation and forced the use of an alternative term to strain, which is displacement/gage length. This limits the comparison between standard samples and samples with holes to be qualitative. Using this term, the effect of exposure time on residual strength is shown in Figure 3 and on the displacement/gage to failure is illustrated in Figure 4 along with data from standard samples as reference. Figure 5 shows the change of the value of strain to failure with the exposure strain for samples with and without holes. It can be seen from Figures 3 and 4 that there is a general reduction in residual strength and residual displacement/gage length with the increase in exposure time and the value of displacement/gage length accumulated during exposure. Standard specimens showed higher residual strength and residual displacement/gage length than specimens with holes. The impact of hole-diameter was not obvious and data for specimens with 2.286 mm holes are at the same level as those from specimens with 4.572 mm holes.

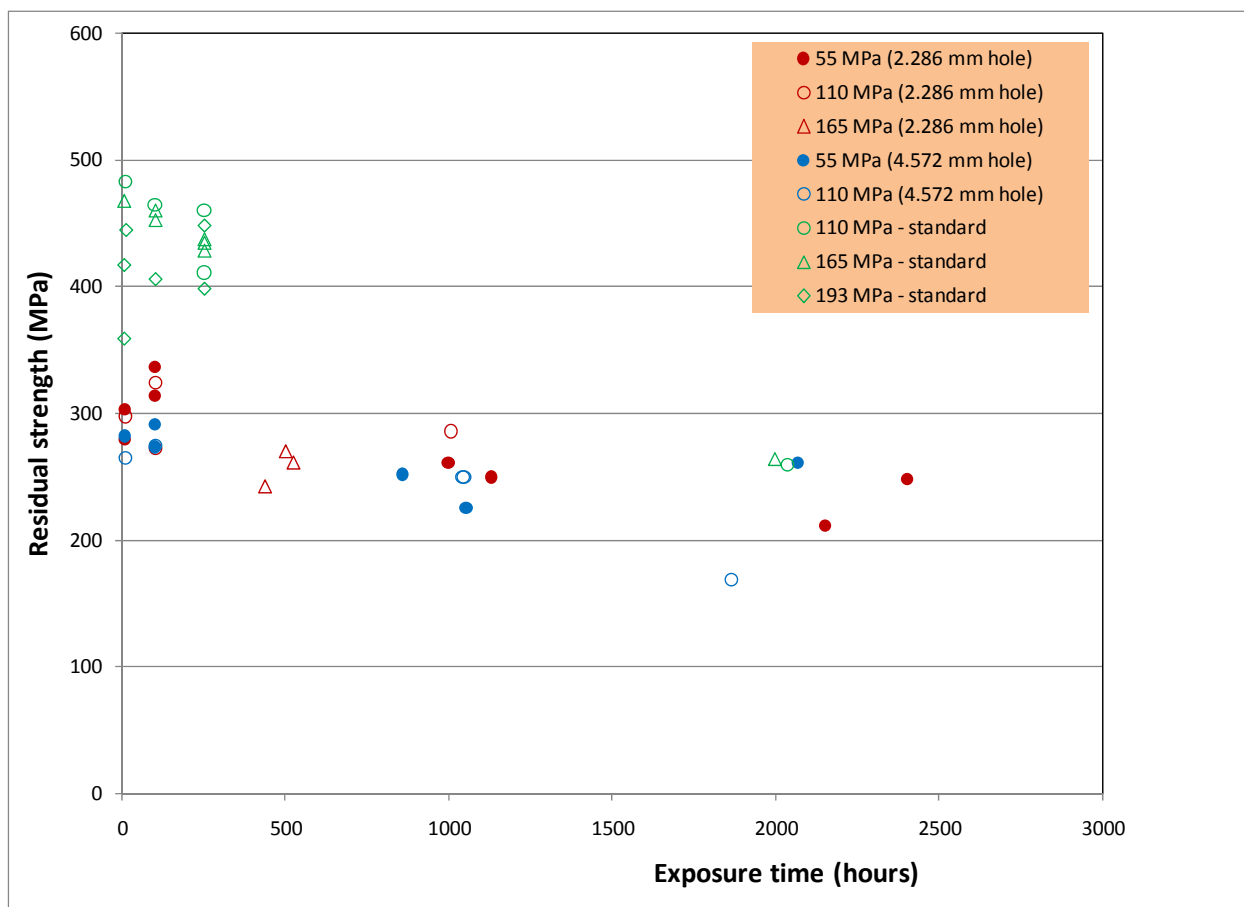


Figure 3. Residual strength for samples with and without holes vs. exposure time for samples subjected to various stress levels during initial testing

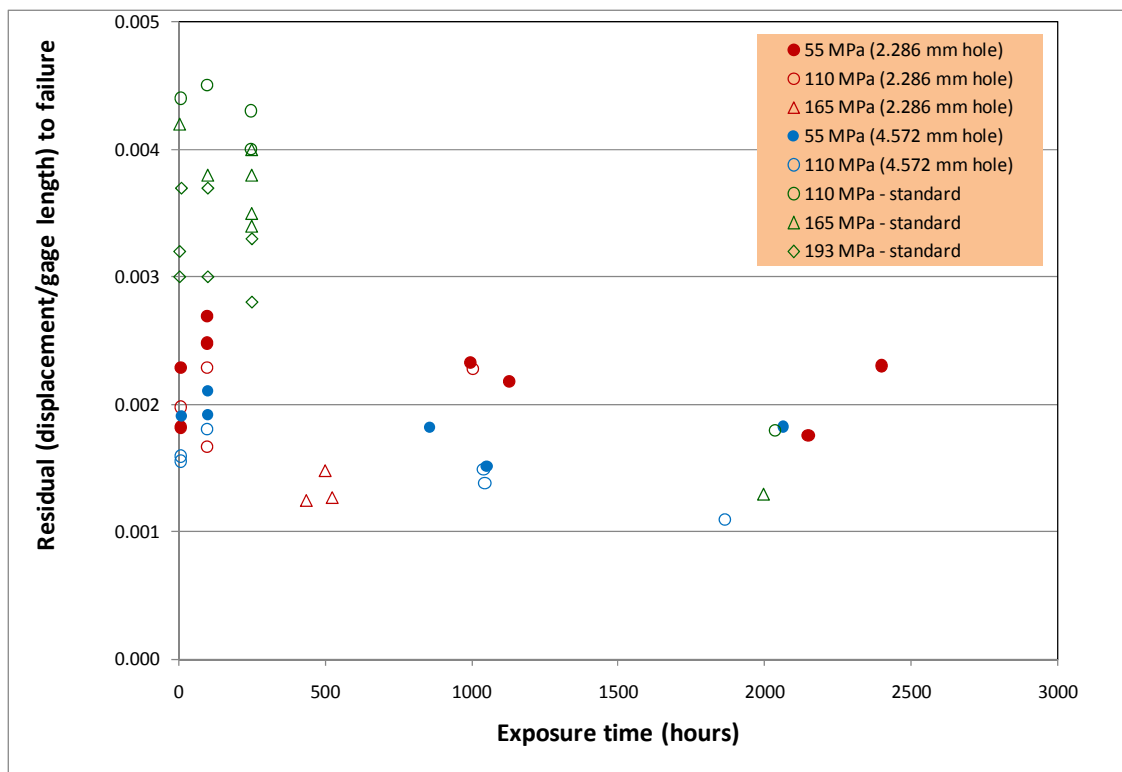


Figure 4. Residual (displacement/gage length) for samples with and without holes vs. exposure time for samples subjected to various stress levels during initial testing

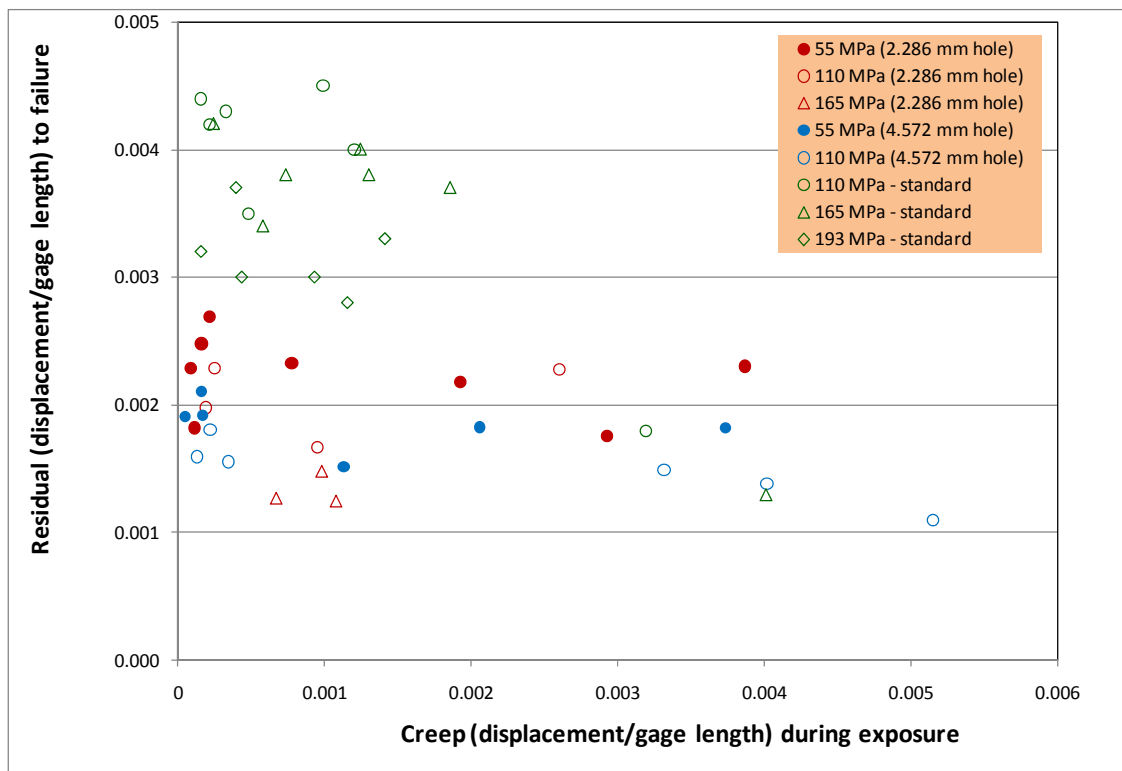


Figure 5. Residual strain for samples with and without holes vs. creep strain during exposure for samples subjected to various stress levels during initial testing

The depreciation of materials capabilities with time-dependent loading under dwell fatigue experiments, as well as fatigue experiments, can be attributed to the reduction in the ability of the fiber/coat and the coat/matrix material to carry shear strength. This concept was studied by Reynaud *et al.* [1,2], and used to characterize the micro-structural origin of the hysteresis by linking it to fiber/coat and matrix/coat interfacial shear assuming that under repeated loading the composite forms a series of interfacial damaged and undamaged zones confined between matrix cracks. Progressive wear at the interfaces between the fiber/coat and the coat/matrix is considered responsible for depreciation of the ability to carry interfacial stresses that starts in limited zones and progresses to cover most of the composite. The approach, as defined by Reynaud [1,2], can be illustrated by two scenarios as shown in Figure 6:

1. Increase in the area of hysteresis loops with the number of cycles leading to a decrease in ability to carry interfacial shear stress (τ) resulting from a local sliding at the fiber/coat or coat/matrix interface.
2. Decrease in the area of hysteresis loops with number of cycles leading to a decrease in the ability to carry interfacial shear stress resulting from a global sliding at all the interfaces.

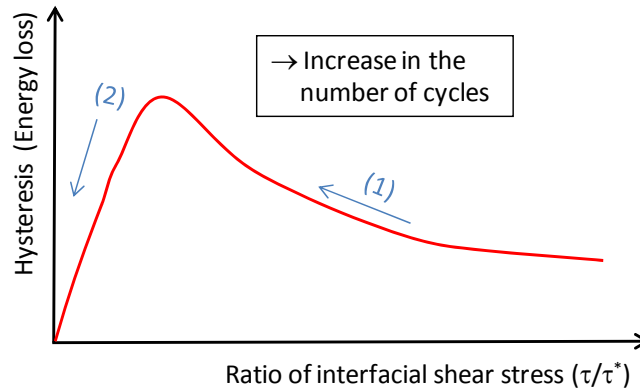


Figure 6: Decrease in interfacial shear stress with mechanical hysteresis following scenario 1 (right) and scenario 2 (left) [1]

As seen from Figure 7, the area of the hysteresis loops decreases with the number of loading cycles following the second scenario indicating a possible decrease in the ability to carry of interfacial shear stress with repeated loading. This can be calculated as follows [1]:

$$\frac{\Delta W}{W_e}(\tau) = \alpha \left(\frac{\tau}{\tau^*} \right) \left(\frac{1 - \frac{2}{3} \frac{\tau}{\tau^*}}{1 + \alpha \left(1 - \frac{\tau}{2 \cdot \tau^*} \right)} \right)$$

Where,

$$W_e = \frac{S}{2 \cdot E_x} ; \quad \alpha = \frac{E_m \cdot V_m}{E_f \cdot V_f} ; \quad \tau^* = \frac{\alpha r E_f S}{2 d E_x}$$

S= maximum stress applied, and r = radius of fibers, ΔW = area of hysteresis loop, d = mean distance between two neighboring cracks, E_f , V_f , E_m and V_m are the Young's moduli and volume fractions of fibers and matrix, respectively, and E_x is the Young's modulus of the composite in the load direction.

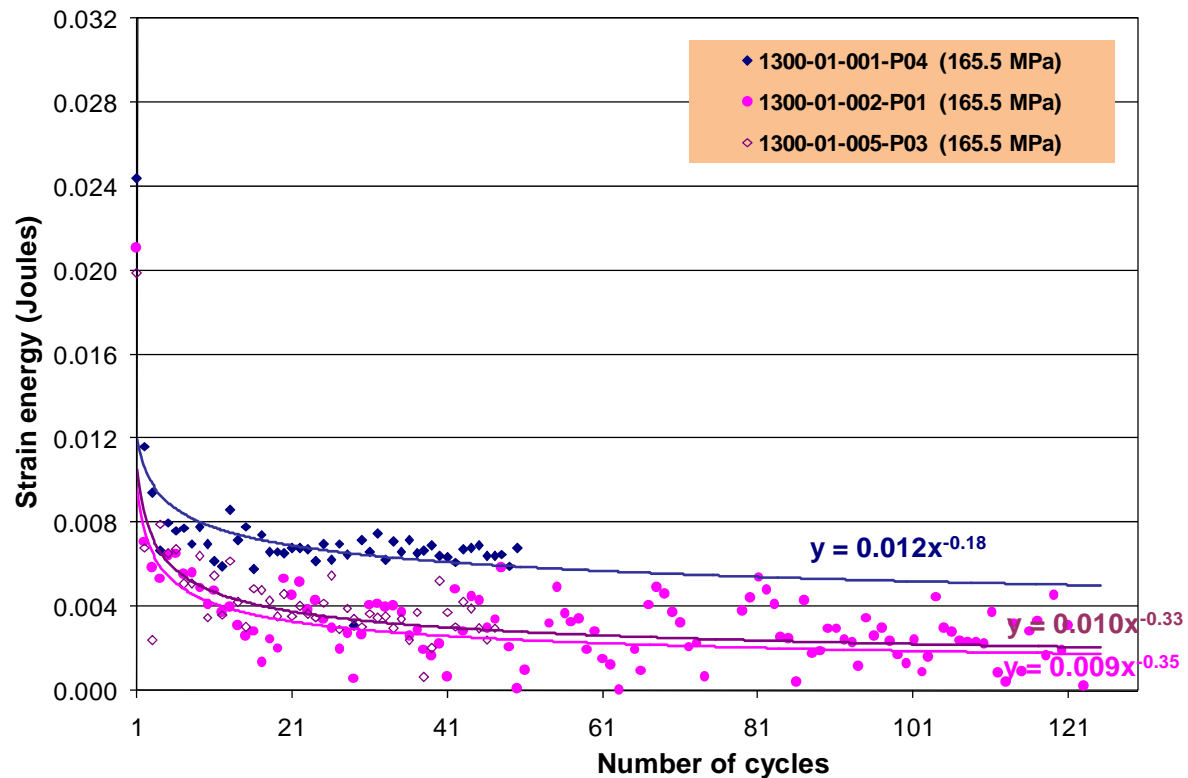


Figure 7. The effect of cycles during dwell fatigue experiments on the area of hysteresis loops of the stress-strain curves

CONCLUSIONS

Residual strength and displacement/gage length to failure of specimens with holes was presented in this work. It was observed that although the effect of the existence of the hole has an impact on the material response when compared to specimens without holes, the effect of the change in the hole size between 2.286 and 4.572 mm did not show a difference in response. A possible damage mechanism due to repeated loading evident in the reduction in the area of hysteresis loops was discussed.

ACKNOWLEDGMENTS

The Materials & Manufacturing Directorate, Air Force Research Laboratory under contract F33615-03-2-5200 and contract F33615-01-C-5234 sponsored this work

REFERENCES

1. Reynaud, P., Rouby, D. and Fantozzi, G., "Effects of temperature and of oxidation on the interfacial shear stress between fibres and matrix in ceramic-matrix composites", *Acta mater.*, vol 46, No. 7, pp 2461-2469, 1998
2. Reynaud, P., "Cyclic fatigue of ceramic-matrix composites at ambient and elevated temperatures", *Composites Science and Technology*, vol. 56, pp. 809-814, 1996.